Ultrathick Photoresist Processing

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Abstract

Diazo-type positive photoresists are commonly used for pattern replication by the integrated circuit industry in a thickness range $\approx$ 0.3-3μm. We are using these same resists at thicknesses as great as 40μm to form electroplating molds for the fabrication of micro-Fresnel zone plates. Difficulties are encountered when films thicker than 15-18μm are used for pattern replication. Most significant of these difficulties are: i) the occurance of bulk microfractures throughout the resist volume, ii) loss of UV sensitivity, and iii) sidewall taper in high aspect ratio structures. These difficulties, with the exception of the sidewall taper, can be overcome with appropriate resist processing schedules.

Introduction

Ultrathick photoresist is being used as an electroplating mold in the fabrication of free standing, gold Fresnel zone plates. The zone plates are used in coded imaging experiments to diagnose the x-ray and particle emissions from laser produced plasmas.[1] Figure 1 illustrates the zone plate geometry. It is characterized by the number of zones
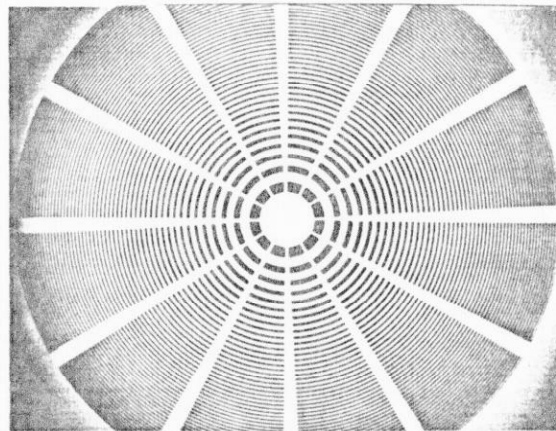

Figure 1. Zone plate photomask with 100 zones and an outer zone width of 5μm.

(n), the width of the outermost zone (Δr_n), and the material thickness (t). The features of a "geometric" zone plate[2] are related by the following expression:

$$r_n = r_1\sqrt{n} \quad (n = 1, 2, 3, 4,...)$$

where r_n is the outer radius of the nth zone.

When used in coded imaging experiments, the attainable spatial resolution of the zone plate camera is proportional to the minimum zone width (Δr_n), the tomographic resolution is inversely proportional to the number of zones (n), and the energy of the x-rays or particles that can be imaged increases with increased zone plate thickness (t). Ceglio and Smith have discussed the use and fabrication of gold zone plates with thicknesses up to 7μm.[3] Because of the need for a high energy x-ray imaging capability, we have extended these techniques to fabricate gold zone plates as thick as 38μm, providing an imaging capability to ~ 40keV. Table I provides a listing of parameters (n, Δr_n, t) for free standing, gold zone plates routinely produced in our laboratory. Figure 2 is a SEM micrograph of a gold zone plate that is 12μm thick, has a minimum zone width of 5μm and is comprised of 240 zones. The radial struts are used solely for

TABLE I

DIMENSIONS OF FABRICATED ZONE PLATES

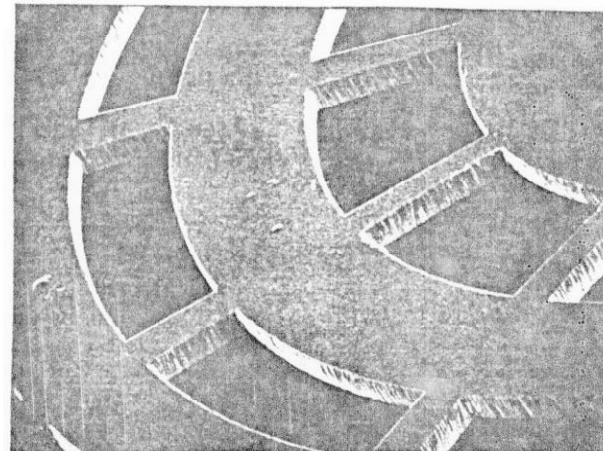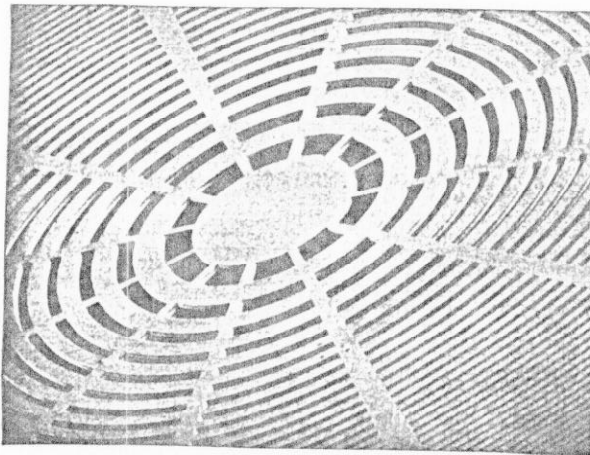| NO. OF ZONES	DIAMETER	OUTER ZONE WIDTH	THICKNESS	THICKNESS/ WIDTH RADIO
n	d(mm)	$\Delta r(\mu m)$	t(μm)	t/Δr
100	0.4	1.0	2	2.0
100	1	2.5	6	2.4
100	2	5.2	(3-10)	(0.6-2.0)
240	5	5.2	(7-18)	(1.3-3.6)
240	10	10.4	(24-36)	(2.4-3.6)
240	14.4	15	(28-38)	(1.9-2.5)

Figure 2.   SEM micrographs of a 12μm thick gold micro-Fresnel
zone plate. Δr_n = 5μm, n = 240.

structural support. An illustrative outline of the procedure used for the fabrication of thick zone plates is presented in Figure 3. Essential to the success of this procedure are steps 2-4, processing the ultrathick photoresist layer, which is subsequently used as an electroplating mold. We have investigated the problems and limitations of ultrathick positive photoresist processing and have developed techniques for the replication of microstructure patterns in 40μm thick photoresist films. In the following sections we detail the processing techniques which have been developed, while discussing the problems and limitations incurred.

Processing Problems

Pattern replication in positive photoresist is usually carried out in thin films (0.3-3μm). The processing details and procedures for this range are well established. Because of our need for narrow linewidths (1-10μm) in our thick micro-Fresnel structures we have pursued pattern replication in these same positive photoresists. Negative photoresists were not found suitable for our purposes because of their lower contrast ratio which results in lower aspect structures and because of their tendency to swell during development which could cause adjacent bars of resist to touch and bridge together.

We have found significant changes in the processing requirements as photoresist thickness increases. There are difficulties incurred with ultrathick photoresist films that are either non-existent or of minor consequence for thin films. This is not surprising since important photoresist parameters such as:
- UV absorption length (ℓ_a)
- Generated gas (N_2) diffusion length (ℓ_d)
- "Diffraction length" (a^2/λ)

do not scale with the increase in photoresist thickness. As a result, when the photo-resist thickness, $\tau_r \gg \ell_a$, extremely long exposure times are required and non-

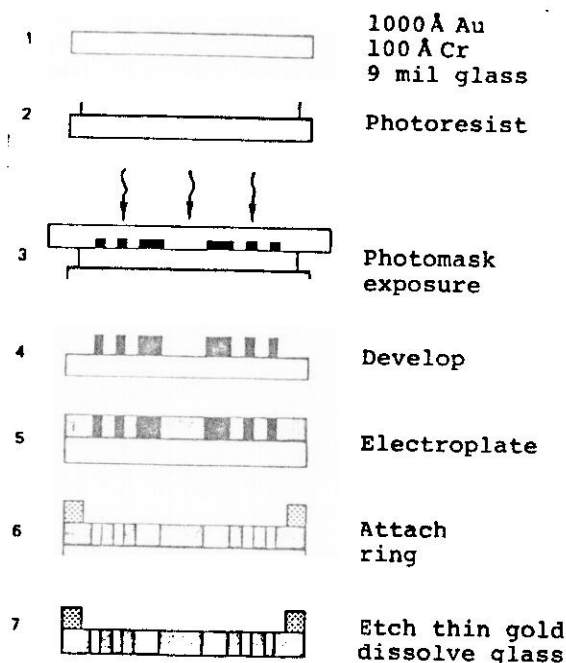|	
1	1000 Å Au 100 Å Cr 9 mil glass
2	Photoresist
3	Photomask exposure
4	Develop
5	Electroplate
6	Attach ring
7	Etch thin gold dissolve glass

Figure 3. Zone plate fabrication sequence.

uniform photoresist exposure may result. When $\tau_r >> \ell_d$, internal stresses can build up within the bulk of the photoresist during UV exposure as evolved gases must traverse a long diffusion path in order to escape (See Appendix). And when $\tau_r >> a^2/\lambda$ diffraction spreading of the UV light within the thick resist layer affects the geometry of the thick resist pattern producing tapering of the resist sidewalls (a = pattern linewidth, λ = UV wavelength).

In developing successful processing schedules for photoresist film thicknesses in the 7-40μm range we identified a number of specific problems. Some of these were minor and perhaps best classified as mere inconveniences. Examples are: resist thickness non-uniformity and irreproducibility; long exposure time requirements; and brittleness of thick resist patterns. Other problems were major, requiring significant changes in processing schedules and careful control of same. In particular, we observed the occurance of microfractures throughout the volume of thick resist layers during UV exposure. This phenomenon could be controlled by extended prebake which in some cases led to a serious reduction in photoresist sensitivity unless followed by appropriate humidity conditioning. For ultrathick photoresist patterns (~40μm) a delicate balance between prebake and humidity conditioning schedules had to be maintained in order to achieve success. Finally, some problems - such as sidewall taper - are more fundamental and require a change in UV wavelength or pattern linewidth (i.e. reduction in structure aspect ratio) for abatement. In the following paragraphs, we discuss in detail each of these classes of problems describing their occurance, possible causes, and procedures used for their control.

Minor Problems

Resist Thickness Irreproducibility and Non-Uniformity. This problem is related to the method of resist application. Ideally, the resist film should be flat so that there are no gaps between the photomask and resist during contact printing. It is also desirable to be able to reproduce a given thickness time after time. There are several methods that can be used to apply photoresist films to a substrate: 1) spinning, 2) spraying, 3) dipping and 4) roller coating.[4] When using any of these application methods for ultrathick films, it is necessary to use multiple coatings of viscous photoresist. Unfortunately, successive coatings dissolve previous layers even when short prebakes are used in between. This results in a buildup of the resist thickness that is not linearly related to the number of coatings applied. In addition, the thickness uniformity over one sample can vary widely if the application method is not closely controlled. We chose to spin on our resist coatings and found it important to not allow the resist to sit on a previous layer for more than a few seconds, and to spin each coating for a full 30 seconds. This method maintained a thickness uniformity per sample to within ± 10% and a batch to batch uniformity also within ± 10%.[6] As a matter of procedure, all thick photoresist films undergo profilometer measurements of thickness following the prebake

and prior to exposure.

Resist Brittleness. Positive photoresist is found to be quite brittle following prebake. This brittleness, a result of the mechanical and thermal properties of the Novalak resin making up the positive resist, will cause it to crack if subjected to undue thermal or mechanical stress. Figure 4 shows a positive photoresist film that was
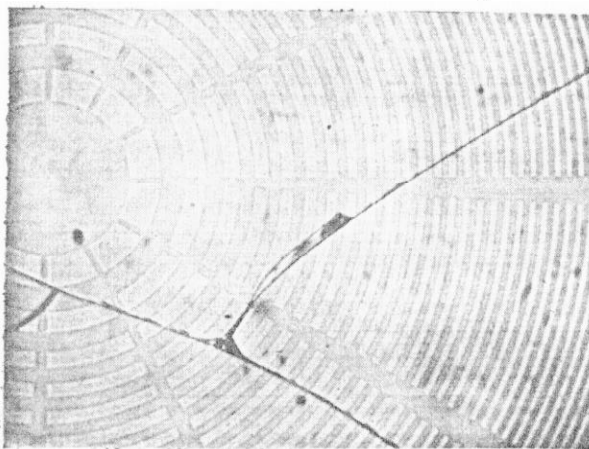

Figure 4. Mechanical and thermal stresses can cause ultrathick positive photoresist to crack. The bleached image of the zone plate is visible in the photoresist.

intentionally subjected to thermal stress following exposure. The long narrow cracks in the film can be seen along with the bleached image of the zone plate pattern.[7]

We found that for photoresist films thicker than 15-18μm, there was sufficient thermal and mechanical stress in the spray developer technique to produce resist cracking as shown. In addition, resist cracking was found to occur during vacuum contact printing for resist films having severe surface non-uniformities (i.e. high spots). The mechanical stress associated with the vacuum contact photomask would cause the resist layer to shatter during or prior to UV exposure.

Photoresist cracking, once it occurs renders a sample virtually useless. Nevertheless, the problem is easily controlled through the use of immersion (rather than spray) development procedures, and proper screening of photoresist samples to reject those with undue surface roughness.

Long Exposure Time. It is not uncommon for pattern replication in ultrathick photoresist to require UV exposure times on the order of one hour. Such long exposure times, while only a minor inconvenience for our zone plate application, could represent a serious limitation to production rates in an industrial environment. Since for the Diazo-type positive photoresists the UV absorption length is of order 1μm, UV energy is initially absorbed only near the top of the film. Only when the photoactive part of the photoresist in this thin layer is bleached can the UV penetrate to underlying resist layers. (The photoresist resin, which is not bleachable only contributes slightly to UV absorption).[8]

Models have been proposed which adequately describe the exposure and development schedules for positive photoresists. In such models the UV sensitive photoactive compound is considered to be an inhibitor which prevents the alkaline developer from dissolving the photoresist.[9] When the inhibitor absorbs UV, it undergoes a chemical change leading to the formation of an acid which is soluble in the alkaline developer. Quantitatively the destruction of the inhibitor by UV light can be expressed:

$$I(x,t) = \exp - [Bx + A \int_0^x M(x',t)dx'] \qquad (1)$$

$$M(x,t) = \exp - \int_0^t CI_o I(x,t')dt' \qquad (2)$$

where:

$I(x,t)$ = UV intensity (normalized to 1)
$M(x,t)$ = fractional inhibitor concentration
I_o = incident UV intensity
x = distance into the film
t = exposure time

A = inhibitor absorption coefficient (μm^{-1})
B = resin absorption coefficient (μm^{-1})
C = inhibitor decay rate per unit
    intensity (cm^2/mj)

Equations (1) and (2) have been solved by Dornberger et. al. for photoresist thicknesses up to 2μm.[10] We have used numerical integration to extend those solutions to thicknesses up to 100μm with an incident UV intensity of 10mω/cm^2 at $\lambda \sim 4047$Å. The following values were used for the coefficients: A = 1.055μm^{-1}, B = 0.094μm^{-1}, C = 0.02cm^2/mj for the Shipley AZ resist and A = 1.184μm^{-1}, B = 0.069μm^{-1}, C = 0.17cm^2/mj for the Kodak 809 resist.**[11]

The results of these calculations are presented in Figures 5 and 6. Plotted are

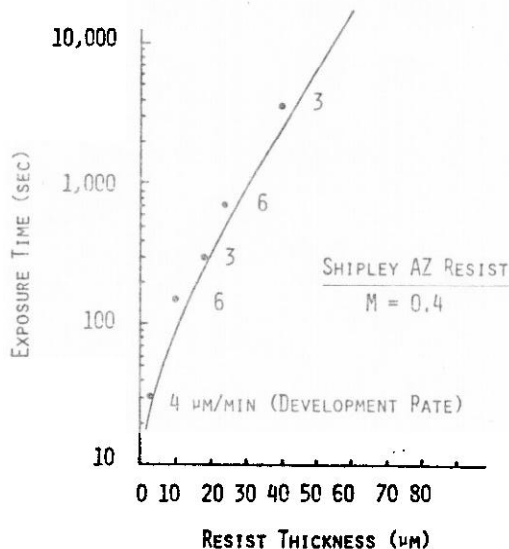Figure 5. Calculated exposure time required for Shipley AZ resist. (I_o = 10mω/cm^2). The curves for M = 0.6 and 0.8 are nearly coincident with the above curve for M = 0.4. Several experimental points are also plotted.
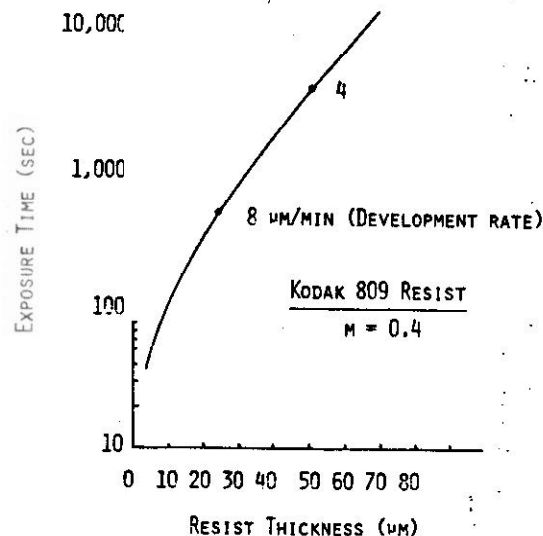
Figure 6. Calculated exposure time required for Kodak 809 resist. (I_o = 10mω/cm^2). The curves for M = 0.6 and 0.8 are nearly coincident with the above curve for M = 0.4. Two experimental points are plotted.

exposure time versus resist thickness for inhibitor concentration reductions to 0.4 for two positive photoresists, Shipley AZ and Kodak 809. (A photoresist with inhibitor concentration of 0.4 is considered to be heavily exposed.)[12] Note that a 50μm film of the Shipley AZ resist requires an exposure of ~ 2 hours, using a 10mω/cm^2 UV source while the same thickness of Kodak 809 requires an exposure of ~ 1 hour. This is due primarily to the higher resin absorption coefficient (B) of the Shipley AZ photoresist. Several experimental points are plotted in Figures 5 and 6 and the observed development rates are indicated. These rates, in the range 3-8μ/min. are in agreement with results reported by others.[11]

Major Problems

Microfractures. Pattern replication in photoresist films having thicknesses in the range 7-15μm can be successfully achieved by following a processing schedule which includes a final 30 minute prebake at 90 °C just prior to UV exposure. However, when photoresist thickness is increased to greater than 15-18μm serious problems are found to arise during UV exposure. These problems take the form of microscopic "bubbles" or "fractures" occuring throughout the volume of the photoresist. Figure 7 is an optical

** Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Department of Energy to the exclusion of others that may be suitable.

TABLE II

PHOTORESIST COATING PROCEDURE

THICKNESS	VISCOSITY	# OF COATS	RPM
3um	31cs	1	1500
6	94	1	1000
10	31	2	1000
15	94	2	1000
21	94	3	1000
30	94	4	1000
42	94	5	1000
54	94	6	1000

Table III and plotted in Figures 5 and 6. A UV source intensity of $10m\omega/cm^2$ was used

TABLE III

EXPOSURE-DEVELOPMENT SCHEDULE SHIPLEY AZ RESIST

THICKNESS	EXPOSURE	DEVELOPMENT
3µm	0.5 MIN	0.75 MIN
6	1.3	1.2
10	2.5	1.8
18	5.0	5.5
24	12.0	4.0
24 (KODAK 809)	8.0	3.0
29	30.0	7.0
40	60.0	12.0
50 (KODAK 809)	60.0	12.0

for exposure. This UV source was a mercury short arc lamp with emission lines at 365nm, 405nm, and 436nm. The intensity was measured with a sensor whose peak response was centered around the 405nm emission line where the photoresist is most sensitive. An "adequate" exposure, defined as one producing an average development rate of 3-8µm/min, was used. Photoresist films less than 18µm thick were spray developed, while thicker films were immersion developed to avoid photoresist cracking. Following development the high aspect ratio photoresist patterns are ready for use as electroplating molds for gold Fresnel zone plate fabrication.

Summary and Conclusions

The need for coded apertures for high energy x-ray imaging has motivated the development of methods for pattern replication in ultrathick positive photoresist. We have identified a number of problems associated with the use of thick layers of the positive photoresists used by the integrated circuits industry, and have developed a series of specific processing procedures for films in the 7-40µm thickness range. We were limited to $\tau_r < 40µm$ by the formation of microfractures within the bulk of the resist. Photoresist patterns greater than 40µm thick will require replication methods other than UV lithography. Two approaches appearing to have promise are x-ray lithography and reactive ion beam etching.
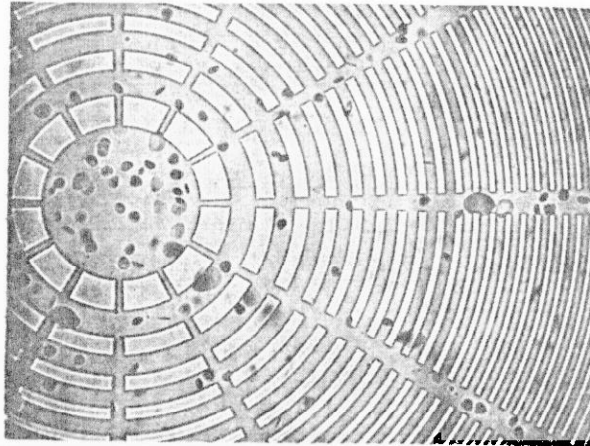
Acknowledgments

Figure 7. Photograph of 25µm film of resist following UV exposure.
Microfractures form in the heavily exposed areas of the
film. The bleached image of the zone plate is also
visible.

micrograph exhibiting the microfracture problem in a UV exposed sample of 25µm thick
photoresist. (The zone plate pattern is bleached into the photoresist by the UV
exposure). Photoresist samples which develop microfractures during UV exposure cannot be
used for high precision pattern replication. They are highly unstable during development
and linewidth accuracy cannot be maintained.

The microfracture problem can be controlled in photoresist films 15-40µm thick by
careful manipulation of the prebake schedule. It was found, for example, that for
photoresist films ~18-24µm thick a final prebake for 16 hours at 70 $^{\circ}$C would eliminate
the occurance of microfractures during UV exposure. This schedule had to be altered for
photoresist films ~24-40µm thick. For such films strict adherence to the following
schedule was necessary to eliminate the occurance of microfractures: final prebake for
10 hours at 85 $^{\circ}$C, followed by a controlled humidity environment for 3-5 hours at
45-50% relative humidity at 22 $^{\circ}$C, followed by immediate UV exposure. The controlled
humidity environment was found necessary to regain resist UV sensitivity after the long
85 $^{\circ}$C prebake. However, increases in the time or humidity of the controlled humidity
environment would cause the microfractures to reappear during UV exposure.

For photoresist film thicknesses in excess of 40µm the delicate balance between
extended prebake to eliminate microfractures and controlled humidity environments to
regain resist sensitivity became very difficult to maintain. Success rates for pattern
replication dropped below 5% and results were in general non-reproducible. A detailed
parametric study of the causes of microfracture formation in ultrathick photoresist films
has not been undertaken. However, the resist behavior as described in the preceding
paragraphs is not inconsistent with what is already known about the properties of
positive photoresist. The evolution of nitrogen during UV exposure of positive
photoresist is well known.[13] It is possible that for a resist thickness sufficiently
greater than the diffusion length for the evolved nitrogen, gas buildup produces sizeable
internal stresses that lead to the formation of microfractures. (A short Appendix is
devoted to a discussion of this mechanism.) Extended prebake schedules may serve to
control microfracture formation either by reducing the rate of nitrogen evolution during
UV exposure or perhaps by strengthening the photoresist making it less susceptible to
rupture. However, the possible affects of trapped solvents on microfracture formation
should not be discounted, although a precise mechanism has not been identified. The
importance of H_2O in the photo-chemical reactions that take place during UV exposure is
well known.[14,15] So then, the importance of humidity conditioning to replenish moisture
content (which had been driven off by an extended prebake) and thereby regain UV
sensitivity is consistent with our expectations of positive photoresist behavior.
Finally, the loss of UV sensitivity in positive photoresists as a result of extended,
high temperature prebake is also well known[12] and could explain the unrecoverable loss of
UV sensitivity in some of the films we worked with.

Sidewall Taper. In the fabrication of high aspect ratio ($\equiv$ line height/line width)
microstructures, the verticality of the pattern sidewalls is a carefully monitored
parameter. Although sidewall verticality is not critical to the proper functioning of
thick micro-Fresnel zone plates in coded imaging applications,[16] it may be important in
other applications of high aspect ratio structures and the affect of resist thickness on
this parameter is worthy of discussion.

Militating against the maintenance of sidewall verticality in photolithographically pro-
duced structures are diffraction effects which spread the UV light after it passes through
the narrow linewidth photomask. For pattern replication in thin resist ($\tau_r << a^2/\lambda$)
diffraction effects can be controlled by maintenance of intimate contact between the
photomask and resist surface. Various techniques have been developed to enhance
photomask contact and thereby improve pattern linewidth control and sidewall taper.[17]
However, pattern replication in ultrathick photoresist ($\tau_r \sim a^2/\lambda$) will suffer from
diffraction effects even when ideal photomask contact is achieved. The UV light is
diffracted into the thick resist layer producing a complicated, three dimensional pattern
of exposure. The ultimate geometry of the resulting resist structure will depend on this
three dimensional exposure pattern in addition to development effects such as the
nonlinear dependence of resist development rate on exposure level and the variation of
contact time (between developer and resist sidewall surface) with depth in the resist.
Detailed computational models exist which, taking into account all these effects, can
provide accurate estimates of the ultimate resist geometry under these conditions.[18]
However, such an analysis is beyond the scope of this presentation. Our message here is
simple and twofold: First, we observe experimentally for photoresist thicknesses
$\tau_r \gtrsim 0.2a^2/\lambda$ sidewall taper becomes a significant problem. Secondly, the problem of
sidewall taper is fundamental, associated with the physical effects of diffraction, and
cannot be mitigated significantly by the mere alteration of resist processing schedules.
In order to increase resist pattern thickness while maintaining sidewall verticality the
choices are simple: increase pattern linewidth, a, or decrease exposure wavelength, λ,
(e.g. x-ray lithography),[19] or, elect an alternate pattern replication process (e.g.
reactive ion beam etching).[20]

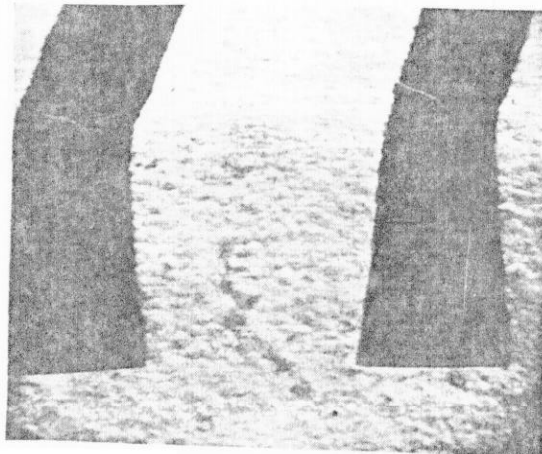Figure 8 illustrates a 24μm thick gold Fresnel zone plate which was electroplated in a

Figure 8.   SEM micrograph of a 24μm thick gold micro-Fresnel zone
plate showing a sidewall taper of approximately 85°.
For this sample, $\tau_r = 0.1a^2/\lambda$.

24μm resist mold having some sidewall taper. The sidewall angle shown is 85°. In this
case $\tau_r \sim .1 a^2/\lambda$.

Processing Procedures and Schedules

All of the photoresist films used in this study were spin applied to substrates. When
multiple coats were used, a 15 minute air dry and 15 minute prebake at 90 °C was used
between coats. Table II lists numbers of coats, resist viscosities and spin speeds
necessary to achieve various photoresist thicknesses in the range 3-54μm.

Following resist application and edge bead removal, each sample underwent a final
prebake. Photoresist samples less than 18μm thick required a one half hour prebake at
90 °C. Samples 18-24μm thick required an extended prebake of 16 hours at 70 °C to
prevent microfracture formation as discussed above. Samples 24-40μm thick required a
prebake schedule of 10 hours at 85 °C followed by a 3-5 hour recovery period in a
45-50% relative humidity environment at 22 °C. For samples greater than 40μm thick
microfracture formation (during UV exposure) could not be reproducibly prevented using
simple prebake and humidity conditioning techniques.

Photoresist exposure and development times for a number of thicknesses are listed in

Appendix

Nitrogen Pressure Buildup in Ultrathick Photoresist During UV Exposure. The following brief discussion provides a rather simple model and plausibility argument for the buildup of sizeable internal gas pressure during the UV exposure of ultrathick photoresist. Although this may be a contributing mechanism to the formation of microfractures, a systematic experimental test of this model has not been pursued and other possible causes of the microfractures should not be excluded from consideration.

We consider the UV exposure of a thick photoresist layer after a time sufficient for the photoactive compound to have been bleached down to depth $L >> \ell_a$. At this time the UV intensity reaching depth L is of order $I_o e^{-BL}$ and it is absorbed by the unbleached inhibitor over a short distance of order ℓ_a. Assume that the absorption of each UV photon liberates an N_2 molecule which contributes to a diffusion current (driven by the buildup of the local N_2 pressure) originating at the thin UV absorption layer and traveling through the resist thickness L to its surface. In the steady state, a pressure gradient is required to drive this current of nitrogen molecules through the resist. The resultant pressure gradient is proportional to the particle current and inversely proportional to the diffusion coefficient; estimates[21] indicate that a pressure gradient of 1 atmosphere/μm is required to drive a particle current of $\sim 4 \times 10^{15}$ molecules/cm²sec. The current density arising from UV exposure will be proportional to $I_o e^{-BL}$. We estimate that for $I_o \sim 10 m\omega/cm^2$ and $L \sim 10\mu m$ the resulting steady state particle current would be of order 10^{16} molecules/cm²/sec., and the steady state condition would be established on a time scale of seconds. So then, under these conditions, we anticipate the establishment of an internal nitrogen pressure gradient of a few atmospheres/μm. Over a distance of 4-5μm a pressure increase of 10 atmospheres can easily be achieved (arbitrarily define ℓ_a to be the distance associated with such a pressure increase), and for resist thickness $\tau_r >> \ell_a$ we anticipate that the internal pressure buildup can begin to threaten the physical integrity of the photoresist.

Reference and Notes

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2. M. Young, "Zone Plates and Their Aberrations", *J. Opt. Soc. Am. 62*, 972 (1972).
3. N. M. Ceglio and H. I. Smith, "Micro-Fresnel Zone Plates for Coded Imaging Applications", *Rev. Sci. Instrum., 49* (1), 15, (Jan. 1978).
4. Another approach could be to use a thick dry-film positive photoresist. (See Ref. 5.) However, it is not clear how well such a resist would behave as a mold for electroplating at thicknesses as great as 40μm.
5. R. A. Mueller, "New Positive Dry-Film Resist", *Plating and Surface Finishing*, pg. 13, (Nov. 1979).
6. A high edge bead was found to build up around the perimeter of the resist sample. It was routinely removed by spinning the substrate while spraying the outer edge with acetone from a hypodermic needle.
7. It was found that in some cases such cracks would self heal if the resist was allowed to sit for 2-3 hours.
8. The UV absorption coefficient for the Diazo-type positive photoresist is $1.06\mu m^{-1}$ for the photoactive compound and $0.09\mu m^{-1}$ for the resin at $\lambda = 4047\text{Å}$.
9. F. H. Dill, J. A. Tuttle, and A. R. Neurenther, "Modeling Positive Photoresist", Proceedings of the Micro-Electronics Seminar "Interface '74", San Diego, California, October 1974, Publication No. G-41, pp. 24-31, (Eastman Kodak Co., Rochester, New York, 1974).
10. Ref. 9, pp. 44-54.
11. J. M. Shaw and M. Hatzakis, "Performance Characteristics of Diazo-Type Photoresists Under-e-Beam and Optical Exposure", IEEE Transactions on Electron Devices, ED-25 (4), 425 (April 1978).
12. F. H. Dill, J. M. Shaw, "Thermal Effects on the Photoresist AZ1350J", IBM J. Res. Develop., pp. 210-218, (May, 1977).
13. Ref. 12, pp. 219-226.
14. D.B. Novotny, National Bureau of Standards, private communcation (1978).
15. J. C. Strieter, "The Chemical Behavior of Positive Working Systems", Proceedings of the Micro-Electronics Seminar "Interface '76", Monterey, California, October 1976, Publication No. G-47, pp. 116-122, (Eastman Kodak Co., Rochester, New York, 1976).
16. Sidewall taper in a Fresnel zone plate will have the effect of smoothing the x-ray transmission profile near the edges of the opaque zones. While this will reduce the efficiency of the coded imaging processes it will not affect zone plate resolution or focal length in image reconstruction (because the periodicity of the structure is unaffected by sidewall taper).

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18. Ref. 15, pp. 81-91.

19. H. I. Smith, "Fabrication Techniques for Surface-Acoustic-Wave and Thin-Film Optical Devices", Proceeding of the IEEE, 62 (10), 1361 (Nov. 1974).

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